THE ANALYST.

SEPTEMBER, 1893.

ON THE RESULTS OF THE WORKING OF THE ADULTERATION ACTS IN DUBLIN.

By Sir Charles A. Cameron, M.D.

(*Read at the Meeting, July 18th*, 1893.)

On August 6, 1860, the first general statute for preventing the adulteration of articles of food and drink was passed by Parliament. It provided for the appointment of public analysts, but not for inspectors of food.

The first public analyst appointed under this Act was the late Dr. Henry Letheby; he was elected to that office by the Corporation of London. The next appointment was made by the Corporation of Birmingham, and the gentleman selected by them, Dr. Alfred Hill, is still in office. I was the third public analyst appointed, having been elected by the Corporation of Dublin, on October 3, 1862.

The Act of 1862 was practically inoperative in every place save Dublin. Some years ago, having made inquiries on this subject, I found that there were no prosecutions instituted under its provisions, except in this city. Shortly after I was appointed I suggested that the inspectors of the markets should purchase articles of food on their own behalf, and submit portions of them to me for analysis. For several years they purchased various kinds of food, and when they were found to be adulterated the vendors were prosecuted. The procedure was simple enough. The purchaser informed the vendor that he was about to submit the article purchased to the public analyst, and that the vendor might, if he chose, accompany the purchaser to the public analyst, in order to witness its delivery. On hearing a case under the Act, the justice might cause a portion of the article purchased to be analyzed by a skilful person, for in those days there was no appeal to Somerset House.

On looking into the records of the Corporation of Dublin, I find that in the first year after my appointment there were 46 articles of food and 11 drugs analyzed. Out of 19 specimens of milk examined there was only 1 pure; the others were adulterated with from 20 to 60 per cent. of water. Eight samples of flour were analyzed, and 1 of them was found to be adulterated with potato flour. Out of 9 specimens of bread, 4 were adulterated with potatoes, rice flour, and alum. One

of two specimens of Bermuda arrowroot consisted wholly of potato starch. The only specimen of coffee tested contained 30 per cent. of chicory. Three of the drugs were adulterated. Thus it will be seen that 50 per cent. of the articles examined were adulterated.

In 1870 the fines inflicted on food adulterators amounted to £158 10s. Amongst the persons convicted were ten confectioners. They had sold confections containing poisonous pigments, and terra alba, or white clay. Some of the lozenges sold by these confectioners contained nearly half their weight of clay. One specimen of sugar-stick contained 1 per cent. of red sulphide of mercury. All the yellow confections were coloured with chromate of lead. Since the year 1871 I have never met with adulterated confections in Dublin.

In 1872, the last year the Act of 1860 remained unamended, the number of convictions for the sale of adulterated food was 67, and the amount of fines imposed was £306 10s. During the ten years that the first food adulteration Act was in force in Dublin, there were hundreds of convictions obtained under it, notwithstanding its defects. The articles found to be adulterated were milk, butter, tea, coffee, cocoa, mustard, flour, bread, arrowroot, confections, sugar, rum and wine. The milk was nearly always adulterated, not only with water, but occasionally, though rarely, with other substances, namely, sugar, starchy matter, and salt. The butter contained excessive quantities of water and salt. On one occasion a sample was examined which contained the pulp of swedish turnip; and on another occasion a roll of butter was found to contain a snowball as its core. On several occasions tea was found to be adulterated with exhausted tea-leaves, the want of substance in it being to some extent disguised by the addition of some astringent body, such as catechu. As a rule coffee was mixed with chicory, burnt sugar, roasted grain, ground toast of bread, etc. In 1874 16 persons were convicted for selling adulterated coffee. In two instances the coffee was mixed with 90 per cent. of chicory, and in four other cases with 70 per cent. and upwards. Cocoa was chiefly composed of starch and sugar. Mustard was never found to be pure. It was always found mixed with large percentages of flour or starch, and even sulphate of lime was used to adulterate it. Flour was adulterated with rice and alum, and bread with rice, potatoes and alum. Confections were adulterated with white clay and coloured with chromate of lead, vermilion, Prussian blue, and, though rarely, with arsenical green. On only two occasions was sugar found to be adulterated, and then it was with flour. Rum was bought which, in reality, was whisky mixed with treacle. I frequently met with the spurious wine termed Hambro' sherry. What was sold as French brandy sometimes proved to be corn spirit, flavoured with various essences.

One good clause in the Acts of 1860 and 1872 was repealed by the Act of 1875, namely, that which enabled the justices to advertise a second conviction in the newspapers, or otherwise, and at the expense of the offender. Advertisements of this kind frequently appeared in the Dublin newspapers. In the front page of the *Irish Times* for February 27, 1872, the following advertisement appears, much "displayed" and in large type:

"PUBLIC ADVERTISEMENT.

ADULTERATION OF MILK.
At the Northern Police Court,
On Saturday, the 17th of February, 1872,
John Doyle, Dairyman,
Of 127, Dorset Street,
Was Fined Five Pounds
For Selling Milk
Adulterated with Fifty per Cent. of Water.

And it being his second conviction for a similar offence, the publication of this advertisement was ordered by the magistrate who heard the case. In addition to the above penalty, cost of advertisement to be defrayed by said John Doyle."

After the passing of the Acts of 1872 the adulteration of many articles of food gradually died out. Tea, flour, bread, mustard and whisky are now never found to be adulterated. Coffee is rarely adulterated, though three cases of adulterated coffee have recently been before the courts. The adulteration of milk is very much less now than formerly. Butter, however, has lately been much subject to adulteration, and I regret to say that the adulteration sometimes takes place in Ireland. With the exception of milk and butter, the food sold in Dublin is, with rare exceptions, pure, a fact which undoubtedly is attributable to the working of the Anti-Adulteration Acts.

With the view of comparing the work done in Dublin in reference to carrying out the Anti-Adulteration Acts with that accomplished in other towns, I have ascertained the number of convictions under the Provision of the Sale of Food and Drugs and the Margarine Acts in 49 towns. The results are shown in the following table:

		Food and Drugs Act.		Margarine Act.	
Town.	Population.	Convictions.	Fines, etc.	Convictions.	Fines, etc.
London—			£ s. d.		£ s. d.
Islington ...	319,433	36	47 5 6	Nil	Nil
Saint Pancras	234,437	31	77 9 0	Nil	Nil
Hackney ...	229,531	22	53 12 0	8	8 17 0
Southwark ...	202,537	22	20 12 6	2	9 7 0
Kensington ...	166,321	32	71 9 6	Nil	Nil
Holborn ...	141,544	3	11 0 0	Nil	Nil
Shoreditch ...	124,009	7	19 6 6	Nil	Nil
Paddington ...	117,838	7	9 8 0	Nil	Nil
Chelsea ...	96,272	15	84 16 6	Nil	Nil
Whitechapel ..	74,462	Nil	Nil	Nil	Nil
Croydon ...	32,548	2	6 0 0	Nil	Nil

Town.	Population.	Food and Drugs Act. Convictions.	Fines, etc.	Margarine Act. Convictions.	Fines, etc.
			£ s. d.		£ s. d.
Liverpool... ...	513,790	115	263 18 8	21	23 19 0
Manchester ...	510,998	66	174 3 6	32	43 12 0
Birmingham ...	483,526	32	29 17 0	15	12 0 0
Leeds	375,540	15	42 10 0	3	6 5 0
Sheffield	329,585	20	41 7 9	4	3 8 6
Bristol	223,592	16	59 10 0	4	4 10 0
Bradford	219,262	3	4 5 0	2	2 12 0
Nottingham ...	215,395	5	10 0 0	1	1 10 0
Kingston-upon-Hull	204,750	10	16 12 6	Nil	Nil
Salford	201,508	24	25 11 0	9	7 17 6
Newcastle-upon-Tyne	192,205	7	3 15 0	Nil	Nil
Leicester	180,066	2	1 0 0	Nil	Nil
Sunderland ...	158,642	Nil	Nil	Nil	Nil
South Shields ...	141,493	1	3 0 0	Nil	Nil
Oldham	134,221	3	3 0 0	Nil	Nil
Blackburn ...	122,238	2	Nil	Nil	Nil
Brighton	116,426	5	11 10 0	1	10 0 0
Birkenhead ...	101,264	4	9 10 0	5	9 0 0
Norwich	100,970	Nil	Nil	Nil	Nil
Huddersfield ...	96,599	4	0 13 6	4	1 0 0
Derby	95,908	11	20 9 0	3	14 4 0
Swansea	92,344	12	19 5 0	Nil	Nil
Gateshead ...	88,588	4	5 0 0	Nil	Nil
Plymouth ...	85,610	14	60 6 0	Nil	Nil
Halifax	84,097	2	1 13 6	Nil	Nil
Wolverhampton ...	83,519	2	2 10 0	Nil	Nil
Middlesborough ...	76,293	4	18 18 0	Nil	Nil
York	72,885	2	3 9 6	Nil	Nil
St. Helens ...	68,628	6	5 0 0	11	5 17 6
London City ...	68,345	1	5 6 6	Nil	Nil
Scotland—					
Glasgow ...	669,059	18	38 3 8	26	37 11 0
Edinburgh ...	264,787	8	13 15 0	Nil	Nil
Dundee ...	155,025	Nil	Nil	Nil	Nil
Ireland—					
Belfast ...	265,123	22	26 3 6	9	17 2 6
Cork... ...	75,345	1	0 10 0	Nil	Nil
Limerick ...	37,155	Nil	Nil	7	24 0 0
Londonderry..	33,200	8	7 0 0	Nil	Nil
Totals ...	8,676,913	626	1,328 13 1	167	243 13 0
DUBLIN ...	245,001	96	201 9 6	44	199 10 0

From January 12, 1893, up to July 13 there have been 62 convictions under the Sale of Food and Drugs Act, and the fines were £137. Under the Margarine Act the

convictions were 38 and the fines £179 10.—Total convictions, 100; total fines, £316 10s.

Discussion.

The Chairman (Mr. Hehner) said that there was no one more qualified to deal with this subject than Sir Charles Cameron, who had a very large district under his control. It was a fact to be regretted that Irish butter had more water in it than the butter furnished by other European countries and New Zealand. Irish farmers continually made excuses for this—excuses which might have a certain small amount of foundation, but which he thought mostly had no foundation at all—excuses such as the state of the weather, and the feeding of the cows, and the breed of the cows. But his strong impression was that the real cause was the ignorance of the farmers in the art of making good butter. He took it that butter was one of the most important of the Irish industries, probably the most important of the future if not of the present time. Irish butter had not for many years stood in such high repute in the English market as other butters scientifically made, such as that of Denmark, the most admirable of all. He (the speaker) would urge most strongly on Irish farmers that the butter industry should be brought to a pitch of perfection similar to that which the Danes had managed to reach in the course of a few years. There an intelligently-advised Government had taken the matter in hand, and in a country where the butter industry had been exceedingly small, and which climatically was no better off than Ireland, they had, assisted by Government aid, and by the industry of the people, within a few years created a butter of the most excellent quality. He took it that if the Irish were willing to be helped, and the Government were willing to help them, that could be accomplished in Ireland also; and if that could be accomplished it would be of more consequence to the country than any other measure that could be conceived. As to the Margarine Act, he agreed with Sir Charles Cameron that in England the Act had been badly enforced, and that Dublin was a place where the Act had been enforced with something like rigour. While, of course, this was in large measure due to the energy of Sir Charles Cameron, he must be congratulated on having a Corporation behind him eager to give him advice in the matter.

Mr. Avon desired to know from what source Sir Charles Cameron derived his statistics.

Professor Tichborne differed from the chairman (Mr. Hehner) in ascribing the presence of large quantities of water in Irish butter to ignorance on the part of the farmers. He thought that the farmers quite knew what they were doing; but it was a very false economy. But lately there had been a great improvement, largely due to the starting of co-operative societies in this Country. Of course, under the old system it was impossible for the small farmer, with his small quantities, to equal the quality turned out by the Dutch producers with their high-class machinery and freezing apparatus. But now they had started in the direction of co-operative societies, and, though only two or three years old, they were spreading to an immense extent in Ireland. The result was that the butter had been largely improved in quality;

and the butter merchants themselves were associating together into societies simply for the purpose of estimating the amount of water in the samples which they bought, so that they should not pass on any quality of butter that would contain more than 15 or 16 per cent. of moisture. He had no doubt that in a few years Ireland would be, as it was now beginning to do, competing with the very finest quality of Danish brands.

Sir Charles Cameron said, in answer to Mr. Avon, that the statistics he had compiled were obtained from the town clerks of the various towns included in the table.

SIMPLE APPLIANCES FOR TESTING THE CONSISTENCY OF SEMI-SOLIDS, WITH NOTE ON A NEW METHOD OF EXAMINING BUTTER.

By Charles E. Sohn, F.I.C.

(*Read at the Meeting, July 18th*, 1893.)

The use of the viscosimeter, which measures the rate of flow of a liquid through an orifice, is limited to substances which are either liquid at ordinary temperatures or assume the liquid condition (without undergoing chemical change) when a moderate degree of heat is applied to them. A large number of technical products exist which do not lend themselves to this method of gauging their physical properties; for instance, semi-fluid compositions holding solid matter in suspension, paints, varnishes, and tarry compounds, emulsion-like mixtures of high viscosity, gelatinous substances and semi-solids generally. In testing the consistency of such as these, the following forms of apparatus may be of service.

Legler, some years ago (see *Chemiker Zeitung* 8, 1857; and Benedikt, *Analyse der Fette und Wachsarten*), described an appliance for measuring the consistency of the elaidin obtained from olive-oil by the degree to which a rod sank into the fatty mass in a given length of time. The rod was graduated at its upper portion, and beneath the graduations there was a disc-shaped expansion serving firstly to keep the rod steady in an outer tube through which it passed, and secondly to support a spiral spring which was adjusted in strength, so as to keep the rod at the zero point when not encountering any other resistance. Dr. R. Kissling (*Chemiker Zeitung*, 1890, 1119, and 1891, 298) has described an instrument for testing railway greases and glue. He measures the length of time taken by a rod to penetrate a certain depth of the sample, and uses several rods of fixed diameter and length, but of various weights and materials.

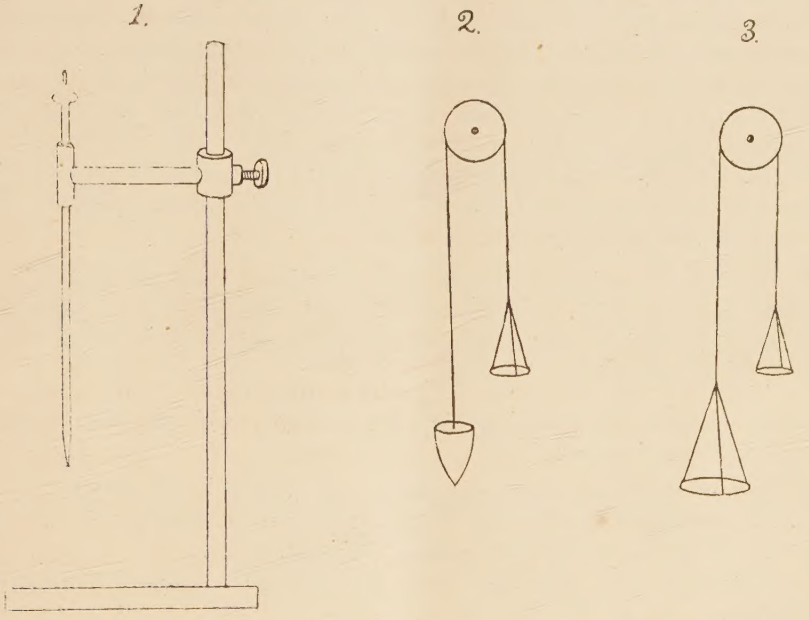
I have employed three different forms of apparatus for consistency tests:

Form No. 1 is really a modification of Dr. Kissling's: Instead of rods of fixed weights, which may be either too heavy or too light for a particular substance, I extend the use of a rod by having it constructed to carry additional weight if required; for this purpose the cap at the upper end has a short prolongation over which centrally perforated coin-shaped weights are slipped. This system of weighting is adopted to

avoid side strain upon the rod, experience having shown that serious error may arise from that cause.*

Form No. 2.—Measurement of the time occupied by a plummet falling a certain depth into the material, the plummet being suspended by a thread running on a pulley wheel, counterbalancing weights being carried by a small scale pan at the other end of the thread.

This form may be employed when the fluidity of the substance is too great for form No. 1; it cannot, however, be used in cases of high consistency, on account of the limited weight of the plummet taken in conjunction with its comparatively large



sectional area. It has an advantage over the rod form in that precautions for ensuring perfect perpendicularity are avoided, if the plummet before its release is allowed to come to rest after oscillation.

Form No. 3.—Measurement of the time occupied by a disc or pan of fixed diameter in *rising* through a given depth of material, when drawn up by a cord variously weighted and running on a pulley wheel. This form also is restricted to compositions possessing considerable fluidity.

Weights and Dimensions.—In view of the great diversity of substances that such appliances may be required to test, and, in fact, the extreme variations observable between members of one class, or even of one substance under different conditions, it does not appear possible to give dimensions that will be generally applicable. It is better for the analyst to determine for himself, by preliminary rough tests, what will be approximately the most convenient sizes and weights for the material in hand.

* The rod glides in a perpendicular tube at the end of a horizontal arm which, like an ordinary retort ring, is adjustable at any height upon a fixed upright.

I have used a brass rod (for form No. 1) of 5 mm. diameter, 300 mm. in length, and weighing 25 grammes, the added weights being 5 grammes to 250, according to circumstances. Dr. Kissling employs rods of glass, zinc, and brass of 50, 100, and 150 grammes respectively, the diameter being 10 mm.

Comparison of Results.—It must be remembered that results obtainable by one apparatus are not readily convertible, by calculation, into terms of another; for instance, if one experimenter employ a rod of 5 mm. and another of 10, not merely will the weight in the second case be distributed over four times the sectional area, and the surface of friction (around the sides) increased fourfold as the rod descends into the material, but the length of time taken to penetrate to a certain depth will probably be greater still than is to be accounted for by these differences, inasmuch as a somewhat limited diminution of weight or increase of surface is frequently sufficient to bring the rod to a stop altogether. Then, again, a rod loses a certain amount of its weight on sinking into the material to a given depth, this loss varying with the specific gravity of the rod and of the material, and being quite different when a hollow rod weighted at the top is used, than when a rod whose weight is equally distributed throughout its length is employed.

In selecting the weights to be used, my experience leads me to believe that the best results are obtainable when the material is penetrated in a comparatively short time—20 to 100 seconds—and that by reducing the weight on a rod so as to extend the time, the accuracy is not increased.

Rules.—Unless the following rules are carefully observed, very erroneous conclusions may be arrived at:

1. The rod must descend in an absolutely perpendicular direction.
2. It must slide in its bearing with the least possible friction.
3. Conditions of temperature must be constant.
4. Vessels of one diameter must be used to contain the material under examination.
5. The rod must enter the centre of the vessel or at a fixed distance from the circumference.
6. The same depth of material must always be used.
7. The material must be allowed to rest a certain fixed time before testing.

With respect to this last rule, Dr. Kissling has suggested a period of two days, but for many reasons a shorter time is more convenient, particularly if the determination has to be made at a temperature other than that of the air at the time, or if the substance is liable to change on keeping. Of course, the maximum consistency may not be reached when a short interval is adopted, but for technical or purely comparative purposes a short period is quite admissible in many cases.

A New Butter Test.—Whilst engaged upon tests with apparatus such as described, my attention was drawn to the possibility of distinguishing pure butter fat from that adulterated with margarine, by testing the consistency of the clarified fat after certain treatment, such as conversion to elaidin; my experiments were but commenced in this particular direction when the announcement appeared that R.

Brullé (see *Compt. Rend.*, 1893, 116, 1255) had already demonstrated the practicability of such a process. His method is as follows:

Five c.c. of the clarified fat are placed in a shallow basin 7 c.m. in diameter on an oil-bath at 148°; when the temperature of the fat has reached 130°, a pinch of powered pumice and 8 drops of fuming nitric acid are added, the whole mixed and kept heated for twelve minutes; it is then allowed to cool to 21° C., and after one hour tested with the instrument that he terms an "oleogrammeter," which is practically identical in principle with the form No. 1 above; it consists of a movable upright rod gliding in a bearing and surmounted by a table on which weights are placed till the rod sinks rapidly into the fatty mass. Most striking differences are by this means shown between pure butter and margarine; in the former case, a weight of 250 grammes, on an average, was sustained, whilst in the latter 5,000 grammes weight was required, whilst mixtures gave intermediate figures capable of approximately indicating the amount of adulteration.*

It is scarcely necessary to remark that the actual weights borne by the fat would be largely dependent upon the shape of the basin holding it.

Whilst I am thus anticipated in this test, it is of sufficient importance to prompt me to bring it before the notice of the members of the Society of Public Analysts, and though from the recent appearance of Brullé's results I have been induced to bring forward these few notes before the conclusion of the experiments I had in view, I hope that the few words said here may lead to a more adequate treatment of the subject of *consistency* elsewhere.

ON THE DETECTION OF HEATED COTTON-SEED-OIL IN LARD.

By William Gustavus Crook.

(*Read at the Meeting, July 18th*, 1893.)

The following is a method for detecting *heated* cotton-seed-oil in lard:

Place about ten grains of the sample to be examined in a cup-shaped porcelain capsule of about half an ounce capacity. A small disc of white filter paper (which has been soaked in hydrochloric acid, thoroughly washed with distilled water, and dried) is *just moistened* with a 12 per cent. solution of nitrate of silver, and placed in the concave part of a watch-glass, which, with the paper downward, is then inverted over the capsule containing the sample. The capsule is then put in a shallow oil-bath, to which heat is gently applied, until a thermometer in the bath reaches 240° F. The source of heat is then *immediately withdrawn*. I find that if even less than 1 per cent. of heated cotton-seed-oil be present in the sample, a very marked coloration takes place on the disc, varying from a light brown to nearly black. If the sample under examination be pure and fresh, the disc is apparently unaffected.

* Upwards of 200 samples of known composition have been examined by this process in the laboratory of the Société des Agriculteurs de France, under the strict control of a specially appointed committee. In every case the result was in accordance with the known composition of the sample.—A. H. A.

NOTE ON THE ESTIMATION OF CHLORINE IN WATER.

By T. Fairley.

(*Read at the Meeting, July 18th*, 1893.)

In analyzing a mineral water from Harrogate, containing only a small quantity of chlorine, a distinct difference in the amount of chlorine was observed according to the method used. In each case 500 c.c. of water was taken, and in titration with $\frac{N}{10}AgNO_3$ solution, potassium chromate was employed as indicator. Thus the chlorine stated in grains per gallon was:

1·34 by weighing as silver chloride.

1·44 by titration of the water concentrated to one-tenth.

1·66 by titration of the water not concentrated.

(This latter result bears out those obtained by Mr. W. G. Young and others. Analyst, May, 1893.)

On repeating the experiments I came to the conclusion that some constituent present affected the results by titration. As this water is alkaline and contains 1·71 grains of silica in solution, and as I find that soluble alkaline silicates when present in small quantity interfere with Mohr's process for the estimation of chlorine, I am led to believe that in this case an alkaline silicate (or what is potentially the same, a solution of soluble silica in sodium carbonate) is the cause of the difference observed. The water in question has been fully analyzed, and does not contain any other substance likely to interfere with the process of titration.

Methods for the Determination of Boric Acid. A. K. Reischle. (*Zeit. anorg. Chem.*, 1893, iv. 111; through *Chem. Zeit.*)—The author has made a comparative study of various methods of determining boric acid, including the following: 1. Weighing the basic acid as boric magnesium borate and determining the magnesia therein. 2. Ascertaining the amount of decomposition of a weighed quantity of sodium carbonate by the boric acid. 3. Distillation of the boric acid as methyl borate (Gooch's method), and weighing as basic calcium borate. 4. Volumetric methods. 5. Weighing as potassium borofluoride. 6. Volatilization as ammonium borofluoride and estimation by difference.

As a result of his investigation, the author concludes that in all cases where the bases present with the boric acid can be weighed as sulphates, the last appears to be the best plan; the boric acid being volatilized as ammonium borofluoride. The same method can frequently be used for the determination of free boric acid. When this process cannot be carried out, it is advisable not to rely wholly upon the other methods, but to check whichever may be used by the fluoride treatment, determining the total bases to be deducted in any convenient way. B. B.

Contributions to the Chemistry of Indiarubber Surrogates. R. Henriques. (*Chem. Zeit.*, 1893, xvii. 634-638). This important paper, dealing with a subject of which very little is known, is an extension of the author's work on the analysis of rubber goods already abstracted (THE ANALYST, xviii. 13). Rubber surrogates consisting of fatty oils that have been treated with sulphur or sulphur chloride, are known in commerce as "factice," variously spelt "facties," "fastice," or in German "faktis." In this abstract the term surrogate will, however, be employed, as being more generally understood. Two kinds of surrogate are used, the white and the brown, which differ in composition. The former occurs in the form of a light yellow friable elastic mass of neutral reaction, and somewhat pungent oily smell. Water dissolves nothing from it, acids and alkalies have but little action upon it, and it is unaffected by most organic solvents. It is characterised by its high content of chlorine, which is nearly as considerable as the amount of sulphur it contains. This chlorine is not removed by treatment with water nor with acids, whence it is evident that it cannot be present as hydrochloric acid or a chloride, but must exist in combination with the organic matter of the surrogate. Its composition, as well as that of other similar preparations, some used commercially and others prepared by the author, is given in the following table :

	S.	Cl.	H_2O.	Ash.	Fatty Acids.	S in Fatty Acids.	Cl in Fatty Acids.	I Number of Surr.	I Number of Fatty Acids.	Acetyl Number
White surrogate A	6·4	5·0	0·85	0·8	90·45	6·12	0·83	30·9	91·3	—
,, ,, B	6·17	5·86	1·0	6·61	73·58	6·45	0·43	31·0	91·2	—
,, ,, C	8·25	8·88	—	—	—	8·15	—	32·6	102·3	—
Brown surrogate A	15·48	0·7	—	—	—	14·14	nil	42·0	129·0	—
,, ,, B	17·71	0·36	—	—	—	15·20	nil	42·0	125·6	—
Linseed-oil surrogate from fresh oil	9·34	8·84	3·02	nil	79·6	9·88	trace	56·3	160·3	21
Linseed-oil surrogate from oxidised oil ...	4·78	4·85	0·85	nil	81·67	4·06	0·60	52·6	141·2	19·6
Rape-oil surrogate from fresh oil ...	8·28	7·62	—	nil	86·89	8·34	trace	32·5	121·0 101·5	31
Rape-oil surrogate from oxidised oil	6·59	5·95	—	nil	87·95	6·54	trace	26·9	102·8	—
Poppy-oil surrogate from oxidised oil	7·68	7·44	—	nil	74·90	8·32	—	33·6	133·3	—
Linseed and rape oil surrogate from oxidised oil ...	—	—	—	nil	—	—	—	42·8	129·2	—
Castor-oil surrogate with a minimum of S_2Cl_2	4·82	6·70	—	nil	85·35	5·32	0·26	35·2	136·2	—
Castor-oil surrogate with a maximum of S_2Cl_2	10·6	8·95	—	nil	—	—	trace	21·9	147·4 152·1 143·5	105·6
"Soluble castor-oil" surrogate from oxidised cotton-seed-oil	6·23	5·36	—	nil	—	6·44	trace	30·3	91·5	51·3

The determination of the sulphur in the surrogates could not be accurately effected by fusion with carbonate of soda and nitre, and the plan that had been worked out for the determination of sulphur in rubber (The Analyst, xviii., 13) was employed with success. In order to estimate the chlorine on the same portion, the process was modified by the addition of nitrate of silver to the nitric acid used to oxidise the rubber surrogate, thus fixing the chlorine as silver chloride, which was afterwards decomposed by fusion with the alkaline melt and the chlorine determined on one portion, while the sulphur was estimated on another. Volhard's method was found convenient for the former purpose. The fact that a considerable percentage of chlorine is present in these surrogates accounts for the fact that bodies of this description, consisting of sulphurized and chlorinated oils, have a low iodine-absorption, a necessary consequence of their partial saturation by a halogen. When the iodine absorption is determined with the two hours period generally used in Germany, fallacious results are obtained, as the fact that the surrogates are sparingly soluble in chloroform militates against the full absorption of iodine taking place in a short time. By dint of repeated shaking, and allowing the absorption to proceed for twelve hours, the full amount of iodine is taken up, and further contact of the oil and the Hübl-solution causes no further combination. The two commercial varieties of surrogate, A and B, gave the figures 30·9 and 31, values which are noticeably low when the high absorption of the drying oil from which the surrogate is prepared is considered. The cause of the low iodine absorption is the state of approximate saturation of the oil, as explained above. In determining the iodine absorption of substances of this description the precaution must be observed to shake the surrogate solution well with the thiosulphate solution when titrating back the excess of iodine, as free iodine is entangled in the mixture of surrogate and chloroform and obstinately retained. The behaviour of the surrogate on saponification also deserves comment. As has been indicated in previous work, the surrogate is soluble in alcoholic potash, and it is found that the chlorine is eliminated, but that the sulphur is not, so that the fatty acids prepared by decomposing the resulting soap contain nearly as much sulphur as the original surrogate. The quantity of fatty acids is, however, smaller than one would anticipate, even when regard is had to the elimination of the chlorine. It was eventually ascertained that portions of the original fatty acids are present as soluble sulphonic compounds, that can be detected by evaporating the liquor (acid with hydrochloric acid) from the separated fatty acids; when the acid becomes concentrated the solution grows turbid, and then, though not before, sulphuric acid can be recognized. Apparently, therefore, the sulphur chloride used to prepare the surrogate, acting in the presence of oxygen either derived from the air or from the breaking down of a portion of the original oil, converts a fraction of the oil into sulphonated fatty acids, similar to those which exist in turkey-red oil. The occurrence of such bodies is common to all surrogates, but the amount present varies with the mode and circumstances of preparation. The elimination of the chlorine already referred to, by saponification, has the effect of causing the iodine absorption of the fatty acids to be higher than that of the surrogate itself (see table). Another deduction from the figures given above is that the samples A and B are prepared from the same kind of raw material. It may be mentioned that both these are typical English products, that made in Germany being of the same type as C.

The course of the reaction between fatty oils and sulphur chloride, as studied by the author to throw light on the mode of preparation of surrogates, is as follows: When the oil is added to a sufficient amount of sulphur chloride, the liquids mix completely, and a vigorous reaction sets in after a few minutes, much heat being evolved, the mass boiling up, and giving off fumes of sulphur chloride mixed with a little hydrochloric acid and sulphurous acid; the whole mass speedily solidifies to an elastic light yellow substance, which is only slightly sticky, and can be ground up under the pestle. On exposure to the air the substance loses any excess of sulphur chloride and hydrochloric acid it may have retained, and becomes neutral in reaction, perfectly resembling the white surrogate of commerce. If the operation be conducted in the same way, save that the reacting bodies are dissolved in some indifferent liquid, such as carbon disulphide, the progress of the reaction is slower, but the end-product is the same, the only apparent alteration being that it is somewhat porous from the evaporation of the solvent, which leaves it open in texture. If insufficient sulphur chloride be used the reaction is less violent, and a sticky mass results, which will not completely solidify even on prolonged standing. The quantity of sulphur necessary for effecting the solidification of an oil varies greatly with the nature of the oil, but is fairly constant for the same oil, as is shown by the following results:

Oil.	Per Cent. of S_2Cl_2 Insufficient to Solidify.	Per Cent. of S_2Cl_2 Sufficient to Solidify.
Linseed-oil	25 ...	30
Poppy-oil	30 ...	35
Rape-oil	20 ...	25
Cotton-seed-oil	40 ...	45
Olive-oil	20 ...	25
Castor-oil	18 ..	20

From these figures it is plain that the drying tendency of an oil has no relation to the minimum quantity of sulphur chloride necessary to form a solid surrogate. Feebly-drying rape-oil requires less than strongly-drying linseed-oil, and castor-oil surpasses all the others in the small amount of sulphur chloride which it needs to convert it into a solid surrogate. All these surrogates are very sparingly soluble in carbon disulphide, such solubility as has been alleged being chiefly due to the presence of imperfectly converted oil, which is extracted by that solvent. The determination of the requisite quantities of sulphur chloride for the solidification of these various oils having been effected, various experimental surrogates were prepared and analyzed by the same methods as those adopted for the commercial products, and the results recorded in the table already given. None of the products thus obtained gave numbers similar to those characteristic of A and B, but the sample prepared from rape-oil much resembled that marked C, wherefore it appears that rape-oil is the raw material of this commercial variety of surrogate. One of the best means of judging the identity of these surrogates is that afforded by the comparison of their iodine numbers, and in this case the figures for this constant are very nearly identical. As C is a representative sample of white surrogate of German manufacture, it is a fair deduction that most German products of this class are prepared by the treatment of rape-oil. Further investigation was necessary to ascertain the nature of the samples

A and B. From the low content of sulphur and chlorine that distinguishes them from C, it seemed probable that they were prepared from an oil that needed for its solidification not more than 20 per cent. of sulphur chloride. The capacity of oxidised oils for absorbing sulphur chloride was therefore examined. Linseed-oil was heated for some hours to a temperature of 200° to 250° C. in contact with air and the product treated with sulphur chloride, of which it absorbed 15 to 18 per cent. in place of 30 per cent., the amount which it took before it had been oxidized. On raising the temperature to 250° to 300° C., the oxidation proceeded so far that no more than 10 per cent. of sulphur chloride sufficed to yield a solid product. This quantity corresponds with a content of 4·78 per cent. of sulphur and 4·85 per cent. of chlorine in the surrogate resulting. Other oils capable of taking up oxygen when heated, such as poppy-oil and rape-oil, behave similarly, although the percentage of sulphur chloride for their conversion cannot be reduced to the low limit attainable with linseed-oil. The power of absorbing oxygen is not the only property of an oil that influences the amount of sulphur chloride which it can take up. The nature of the glycerides of which the oil is composed is an important factor, as is evidenced by the remarkable figure for castor-oil given in the last table. The reaction between castor-oil and sulphur chloride is so vigorous that it has to be moderated by the use of an indifferent solvent in the manner described above. The surrogates prepared from oxidized linseed-oil were different from samples A and B, being darker in colour, a drawback that could, however, be avoided by conducting the preliminary oxidation at a lower temperature, a process which is now actually in use for the production of pale "boiled" linseed-oil. But the difference is more profound than can be accounted for by a variation in the mode of oxidation, as can be seen by a comparison of the figures in the first table. After some further investigation the author was able to determine the nature of the oil from which these two commercial samples had been prepared. There is an oil known in the trade as "soluble castor-oil," "oxidized castor-oil," and by similar titles, which is of English make, and is used as a lubricating oil. It is in reality blown cotton-seed-oil. This oil unites with 20 per cent. of sulphur chloride to form a solid surrogate, while unoxidized cotton-seed-oil requires about 50 per cent. of the reagent. By preparing surrogate from this material, the substance whose analysis is given at the end of the first table was obtained, and its substantial identity with samples A and B determined. The reasons for the use of this surrogate, rather than the others of German make, are no doubt that sulphur chloride is dearer than cotton-seed-oil, and economy in the former is advantageous, while it is probable that its comparative poverty in sulphur and chlorine makes it a better rubber substitute than the surrogates rich in those elements. In most cases, with the information now at our disposal, the determination of the percentage of sulphur and chlorine in a surrogate and of the iodine number of its fatty acids suffices to indicate the kind of oil from which it has been prepared. In order to ascertain whether it is possible to prepare a surrogate which is saturated with the sulphur and chlorine of sulphur chloride, the author treated castor-oil with a large excess (50 per cent.) of sulphur chloride, and extracted the mass with carbon disulphide, which removed any unchanged oil. The analytical figures given by this substance are recorded in the first table. It will be seen that the iodine absorption has not been

brought to a vanishing point, an indication that complete saturation was not attained. By further purification with glacial acetic acid and aqueous caustic soda the iodine number was reduced to 14, but could not be brought to O. It is not quite certain from this persistent absorption of iodine that the substance still contains unsaturated compounds, as a portion of the halogen may be used up in attacking the sulphur. It is, however, evident that the main action of the sulphur chloride is the conversion of unsaturated bodies into saturated substances, and that unsaturated compounds are regenerated on saponification, as can be realized by comparing the iodine absorption of the original surrogate with that of the fatty acids obtained from it. Generally, the iodine absorption of the fatty acids is nearly equal to that of the oil before it has been treated with sulphur chloride, but castor-oil presents a noteworthy exception to this rule, the iodine number for the fatty acids from the treated oil being greatly higher than that for the untreated oil (83 to 85). This is to be explained by the supposition that the fatty acids recovered from most surrogates are derivatives of the oleic series, while those from castor-oil belong to the oxy-linoleic series $C_nH_{2n-4}O_3$. Additional light on the constitution of these bodies is thrown by their acetyl number. The usual method for determining the acetyl number was not applicable in this instance, as the liquid was too dark coloured to permit of accurate titration with phenolphthalein as an indicator. The modification adopted by the author consisted in decomposing the acetylized acids with caustic potash and distilling with sulphuric acid, the acetic acid being volatilized and determined in the distillate. The plan in the author's hands gives identical results with Benedikt's. Using this process, the number 105·6 was found for the treated castor-oil already spoken of (see table), a figure considerably lower than that given by untreated castor-oil. The deduction from this experiment is that the saponification of the surrogates does not involve the introduction of an hydroxyl group, and that the group (SH) is also absent.

The foregoing work deals with the white surrogates, and a few words must be said concerning the brown variety. The latter are dark-coloured, somewhat sticky, and contain much sulphur and but little chlorine; their price is lower than that of the white surrogates; it is probable that they are made by heating the oil with sulphur, instead of sulphur chloride. These brown products are completely saponified by alcoholic soda, and on liberating the fatty acids from the resulting soap a little sulphuretted hydrogen is given off, while the amount of sulphur in the fatty acids is but slightly less than that in the original surrogate. The iodine absorption of these surrogates is high, whence it appears that they are made from linseed-oil or mixtures of linseed and rape oil. B. B.

The Analysis of Rubber Goods. R. Henriques. (*Chem. Zeit.*, 1883, xvii. 707-709.)—This work is a continuation of that of the preceding abstract. The author has already shown that there are two kinds of commercial rubber substitutes: the white, which is manufactured by treating various oils with sulphur chloride; and the brown, in the production of which sulphur itself is used; and that the former can be discriminated from the later by the presence of chlorine. The quantity of chlorine is not, however, that which might be expected from the percentage known to exist

in the surrogate. Thus, two rubbers containing surrogate, the chlorine in which amounted to 3·7 and 0·9 per cent. respectively, reckoned on the mixture of rubber and surrogate, were found to contain only 0·50 and 0·37 per cent. of chlorine. Apparently, therefore, a large part of the chlorine is eliminated in the vulcanizing, being volatilized either as sulphur chloride or hydrochloric acid. The next point of analytical importance is the detection of fatty oils in the presence of surrogates which have been prepared either by treatment with sulphur chloride or with sulphur. Fatty oils are not appreciably sulphurized during the vulcanizing process, and, as the fatty acids obtained by acidifying the extract obtained by digesting the rubber under examination with alcoholic soda only contain sulphur when they have been derived from sulphurized oil, the percentage of sulphur present gives a measure of the amount derived from fatty oils in contradistinction to that produced from the saponification of any sulphurized surrogate. This view of the matter was put to the test of experiment in the following way: Rape-oil was heated with excess of flowers of sulphur to a temperature of 130° to 135° C. for several hours. A considerable quantity of sulphur dissolved in the oil and crystallized out on cooling. The fatty acids from the rape-oil thus treated were found to contain 0·98 per cent. of sulphur, thus proving that the amount of sulphur taken up by rape-oil at the highest temperature used in vulcanizing rubber is quite small, and could not lead to its confusion with surrogate. It is possible, therefore, to detect any two of the three additions to rubber—viz., white surrogate, brown surrogate, and fatty oil—and to estimate the quantity approximately; but when all three are present (a rare occurrence) the task cannot be satisfactorily accomplished.

The detection of white surrogate depends greatly on the fact that it contains chlorine, while rubber is generally free from this element. An exception, however, occurs in the case of rubber vulcanized by the cold process. Cold vulcanized rubber, so-called "patent rubber," is prepared by treatment with a solution of sulphur chloride in carbon disulphide, and might be expected to contain chlorine. It may be remarked parenthetically that an erroneous idea of what is meant by the technical term "patent rubber" is prevalent and should be corrected. The definition is sometimes found in technical works, that patent rubber is that which has been treated with caustic alkali after vulcanizing to remove the excess of sulphur. This is entirely a mistake. A much nearer approach to a definition would be that patent rubber is prepared from patent plate rubber. Patent plate rubber is not moulded or rolled, but cut from a block in manner similar to the preparation of wood veneer. In order to cut the blocks properly, it was formerly customary to bury them for some months in the earth, but now the necessary increase of firmness and consistency is obtained by artificial cold instead of the natural low temperature of the earth. A characteristic property of patent rubber is its finely-ribbed surface, which can be seen on the black rubber tubing used in the laboratory. The existence of this marking is not, however, a proof that the rubber is genuine patent rubber, as the plan has been adopted of imparting a ribbed surface to inferior rubber, prepared by rolling into sheets by pressing it against a textile material suitably rugose. The main question to be settled, namely, whether patent rubber contains chlorine or not, and whether, therefore, the methods of detecting surrogate that had been devised for

rubber vulcanized in the ordinary way are applicable to it, was investigated in the following manner :

The author vulcanized thin sheets of Para rubber by the cold process, and submitted them to analysis. Sample I. was over-vulcanized, and on that account hard and somewhat inelastic, while Sample II. was under-vulcanized and a little sticky at 100° C. The method of analysis was that described in the author's previous paper (The Analyst, vol. xviii. 113) :

	I.	II.
Ash of raw rubber	0·46%	0·46%
Sulphur	5·19	0·50
Chlorine	5·61	0·57
Extract with alcoholic soda	2·9	2·2
Ash of extracted residue	0·67	0·9

The total quantity of material soluble in alcoholic soda is small, and, further, contains appreciable amounts of sulphur and chlorine, whence it is apparent that rubber vulcanized by the cold process behaves towards alcoholic soda (the best solvent for surrogate) much in the same way as does rubber vulcanized by heating with sulphur. This view was confirmed by the analysis of an authentic sample of patent rubber and of the raw rubber from which it had been made :

	Patent Rubber.	
	Unvulcanized.	Vulcanized.
Ash	0·18%	0·18%
Sulphur	—	1·07
Chlorine	—	0·89
Extract with alcoholic soda	1·94	1·66
S in soda extract (calc. on original rubber) ...	—	0·57
Cl in do. (do. do.) ...	—	0·55
Rubber in do. (do. do.) ...	1·94	0·54

Similar results were expected in ordinary commercial samples of patent rubber, as it is repeatedly stated in technological handbooks that only pure Para rubber can be cut into patent plate. The first sample was one of cheap quality from a North German factory, the two others being samples of good rubber tube of different origin :

	I.	II.	III.
Ash	0·67%	— %	0·2%
Sulphur	3·68	2·37	1·86
Chlorine	3·63	2·51	1·33
Residue after extraction with alcoholic soda	84·3	92·1	94·5
Containing :			
Ash	1·8	—	0·88
Sulphur	1·92	1·77	0·83
Chlorine	1·31	1·54	1·17
Extracted by alcoholic soda :			
Sulphur and chlorine	4·08	1·57	1·19
Organic matter	12·5	6·5	5

In all cases the percentages are reckoned on the original rubber.

In order to ascertain the nature of the surrogate that had been added to Sample I., and had been detected by the foregoing results, a large quantity of the

rubber was extracted with alcoholic soda, the alkaline extract acidulated and the fatty acids removed by solution in ether. They constituted a thick oily residue which gave the following analytical figures :

Sulphur	6·29%
Chlorine...	0·35
Iodine absorption	92

These numbers are almost identical with those previously obtained for white surrogate, and it therefore appears that "patent rubber" occurs in commerce adulterated with vulcanized cottonized or rape-oil. This conclusion, deduced from the figures obtained by analysis, was confirmed by a communicative manufacturer, according to whom patent rubber has been made for a number of years of mixtures containing as much as one-third surrogate, although the practice has not been generally known to exist. The fact that a rubber is sold as "patent rubber" is no longer to be considered as a guarantee of genuineness, even when the mode of preparation is that proper to patent rubber.

B. B.

Hydrolysis of Fats by Concentrated Sulphuric Acid. A. Prager and J. Stern. (*Chem. Zeit.*, 1893, xvii. 880.)—By using sulphuric acid of the exact strength recommended by Kreis (91·53 per cent. H_2SO_4) in his latest communication (ANALYST, vol. xviii. 145), the authors have certainly obtained numbers agreeing better with the Reichert-Meissl numbers than those which they obtained when they followed Kreis' original specification, but *only with pure butter*. A mixture of butter with margarine yields numbers by Kreis' method which are higher than the Reichert-Meissl numbers in proportion to the percentage of margarine present. It is generally admitted that the Reichert-Meissl method will not detect 10 per cent. of margarine in butter with certainty ; much more true is this of the Kreis method. The following figures may be quoted :

	No. of c.c. of $\frac{N}{10}$ Alkali required by 5 Grammes of Pure Butter.					
	I.	II.	III.	IV.	V.	VI.
Reichert-Meissl ...	27·23	30·78	31·79	30·14	30·88	30·15
Kreis	27·84	29·96	32·28	31·18	31·22	31·12

MIXTURE OF BUTTER WITH MARGARINE.

Percentage of Margarine.	No. of c.c. of $\frac{N}{10}$ Alkali required by 5 Grammes of the Sample.	
	Reichert-Meissl.	Kreis.
0 ...	26·05 ...	26·63
10 ...	23·64 ...	24·50
20 ...	21·14 ...	24·53
40 ...	16·27 ...	19·35
60 ...	11·38 ...	16·09
100	0·85	0·98

In the majority of cases the numbers obtained by Pinette's modification were nearly as much lower than those obtained by the Reichert-Meissl method as the Kreis numbers are higher, although in several instances (not here quoted) the numbers agreed fairly well.

Percentage of Margarine.	No. of c.c. of $\frac{N}{10}$ Alkali required by 5 Grammes of the Sample. Reichert-Meissl.	Pinette.	Prager and Stern.
0 ...	30·14 ...	28·78 ...	30·32
10 ...	27·36 ...	25·48 ...	27·40
20 ...	24·64 ...	23·02 ...	24·58
40 ...	19·10 ...	17·35 ...	19·16
60 ...	13·44 ...	11·21 ...	13·56
100 ...	2·65 ...	1·96 ...	2·70

The authors conclude that removal of sulphurous acid cannot be omitted, and must be effected by the method proposed by them. A. G. B.

The Determination of Manganese by Means of Hydrogen Peroxide. A. Carnot. (*Compt. Rend.*, 1893, cxvi. 1375; through *Chem. Zeit.*)—In order to determine manganese by means of the available oxygen of its peroxide, it is necessary in the first place to prepare a peroxide of definite composition. This can be effected in several ways.

In the first method the compound containing manganese is dissolved in strong nitric acid, with the addition of small quantities of potassium chlorate, the manganese being thus converted into MnO_2. The separation of manganese from other metals is complete, save in the case of iron, which accompanies the manganese to some extent, and can be separated by solution of the peroxide in nitric acid with the addition of hydrogen peroxide, evaporation, and reprecipitation with strong nitric acid and potassium chlorate. After thorough washing of the purified manganese peroxide first with cold and finally with hot water, it is determined by measuring the oxygen evolved by treating it with hydrogen peroxide and nitric acid in the manner described below.

Better results than those yielded by the chlorate method are obtained by the use of ammoniacal hydrogen peroxide to bring the manganese to a definite state of oxidation. Hydrogen peroxide in excess is added to the solution containing the manganese, excess of ammonia quickly added, and the mixture boiled for some minutes, whereby a dark-brown precipitate of the composition Mn_6O_{11} is produced. Large quantities (10 to 20 grammes) of ammonium salts, notably, the acetate, hinder the formation of this peroxide, but the difficulty can be overcome by the use of a large excess of hydrogen peroxide. Should the precipitation be effected in the presence of copper, zinc, nickel, or cobalt, the manganese peroxide contains a small amount of the oxides of these metals, which can be removed by several precipitations. Small quantities of iron do not interfere, but when iron predominates a preliminary separation with nitric acid and potassium chlorate is advisable.

The final determination of the manganese is carried out by placing the peroxide, however obtained, in a flask, together with dilute nitric acid, running in a known volume of hydrogen peroxide, and measuring the air displaced by the oxygen evolved. B. B.

Note by Abstractor.—The method is probably feasible, as it has been used by the undersigned for red lead; but as the manganese peroxide is obtained pure, it would be simpler to ignite and weigh as Mn_3O_4. B. B.

The Adulteration of Margarine with Sunflower Oil. A. Jolles and E. Wild. (*Chem. Zeit.*, 1893, xvii. 879.)—Two samples of margarine recently examined by the authors appeared to be adulterated with cotton-seed oil on account of their high iodine absorption and the positive indications given with Becchi's solution and the nitric acid test. A third sample of the same class gave the following figures :

Melting-point of fatty acids	43° C.
Solidifying-point ,, ,, ...	39° C.
Iodine absorption	60·34
Becchi's test	Positive indication
Nitric acid test	Negative indication.

On account of the failure to obtain any characteristic brown colour on shaking with nitric acid (sp. gr. 1·37), the authors considered that some vegetable oil other than cotton-seed oil was present. Such an oil, to be remuneratively used as an adulterant, must be of pleasant flavour and not much dearer than cotton-seed oil, these conditions excluding olive, rape, and linseed oil. Seeing that the iodine absorption of sunflower oil is about 127 and that of cotton-seed oil 107, the figure given above for the suspected sample corresponds with an adulteration of 22 per cent. of the latter and 15 per cent. of the former, whence it follows that cotton-seed oil could not have been present, as it would have been indicated by the melting and solidifying point of the fatty acids, by the elaidin test and by the brown coloration with nitric acid. Pursuing their investigations, the authors found that the difference of behaviour of cotton-seed oil and sunflower oil with nitric acid (of sp. gr. 1·37)—the former giving the brown coloration above mentioned, and the latter showing no change of tint—serves as a criterion even when as little as 10 per cent. of either is present in margarine. A trial mixture containing 85 per cent. of margarine and 15 per cent. of sunflower oil had an iodine absorption of 61·5 per cent., the iodine absorptions of its constituents being 47 and 127 respectively, while a mixture of 90 per cent. of margarine and 10 per cent. of sunflower oil gave the number 55·37, and one of 85 per cent. of margarine and 15 per cent. of cotton-seed oil 55·8. The first result, therefore, corresponds with that of the adulterated sample. The nitric acid test was also effective in distinguishing between these synthetic samples. This test can be conveniently made on a chloroformic solution of the oil.

It is noteworthy that the price of cotton-seed oil has recently risen, so that it is now nearly double that of sunflower oil—a circumstance that gives ground for suspecting the extended application of the latter as an adulterant. B. B.

The Nessler Reaction for Ammonia. B. Neumann. (*Chem. Zeit.*, 1893, xvii. 880.)—L. L. de Koninck has stated (*Zeit. anal. Chem.*, 1893, xxxii. 188) that Nessler solution gives no reaction with ammonia in alcoholic solution. The author has found, on the contrary, that, in alcohol of strength ranging from 1 to 100 per cent., a trace of ammonia gives a definite reaction with Nessler solution, and, in fact, that the influence of the alcohol is almost negligible. He attributes the discrepancy to some want of sensitiveness in the specimen of Nessler solution used by De Koninck.

B. B.

The Examination of Beeswax. G. Buchner. (*Chem. Zeit.*, 1893, xvii. 918, 919).—The best method of examining beeswax is that due to Hübl; but there are many fictitious waxes (*e.g.* such a mixture as 35 parts of stearic acid, 165 parts of japan wax, and 300 parts of paraffin wax) which can be added to beeswax without being detected by this plan, or the melting point, or specific gravity. The author's method of examination is as follows.

The wax is first melted with distilled water, and the resulting cake of wax allowed to cool, removed and dried. Should the water be acid to litmus—which may arise from the bleaching of the wax with acid, followed by imperfect washing—the cake is remelted with water several times, as the presence of acid may affect the determination of the constants of the wax. The final fused product must be clear. The purified wax is examined in the conventional manner.

I. *The wax gives normal figures* (acid number, 19-21; ester number, 73-76; saponification number, 92 to 97; ratio of acid to ester number, 3·60 to 3·84). It may be either genuine wax or a cleverly sophisticated sample. Further tests are therefore necessary.

(1) Stearic acid is looked for by Döttger's modification of Fehling's test. One grm. of wax is boiled for some minutes with 10 c.c. of alcohol, cooled to 18-20° C., filtered, the filtrate treated with water and shaken. Stearic acid separates in flocks and collects on the surface of the liquid, while the underlying aqueous portion becomes clear. One per cent. of stearic acid can be thus detected. Should as much as 7-8 per cent. of stearic acid be present, it remains diffused through the water as a thick creamy mass.

(2) Rosin is looked for by Donath's method, modified by E. Schmidt. Five grms. of wax are treated with 4 to 5 times as much nitric acid of sp. gr. 1·32 to 1·33, and heated therewith to boiling for 1 minute. An equal volume of water is then added, and afterwards ammonia in excess. The liquid is poured off the separated wax, and is yellow at most should the wax be pure, but more or less brown should rosin be present. One per cent. of rosin can thus be detected. A blank test should be made with pure wax.

(3) Glycerides (*e.g.*, japan wax and tallow) are recognized by the isolation of glycerine. For this purpose the residue (still warm) left after the execution of the Hübl method of examination (*v.s.*) is evaporated on the water-bath; the alcohol driven off, water added, the filtrate concentrated and tested for glycerine by heating with potassium bisulphate.

Should these tests give negative results the wax is genuine, seeing that the acid number and other constants are normal. It may be noted that the commonest adulterants are paraffin wax, japan wax, carnauba wax, and stearic acid.

II. *The wax gives abnormal figures.*—In this case the sample can be condemned out of hand, save when the only abnormality is a somewhat higher acid number, and tests for rosin and stearic acid give negative results. The acid number in bleached wax may be as high as 24 without the sample being necessarily sophisticated; otherwise adulteration has certainly been effected. The nature of the adulterant is ascertained by the tests under I., and the quantity calculated from Hübl's constants. In special cases the glycerine present must be determined, and the paraffin wax by

Buirine's method. The author draws attention to the fact that although but little pure beeswax is sold, yet the stuff on the market gives nearly normal figures, a proof that the fraudulent application of chemical knowledge is frequent. B. B.

REVIEWS.

ANALYSIS OF MILK AND MILK PRODUCTS. H. LEFFMANN and W. BEAM. Philadelphia: Blakiston, Son, and Co. 1893. 12mo., 92 pp.

Drs. Leffmann and Beam have recognised the want of a manual on milk analysis and have produced what must be pronounced the best book on the subject in the English language; for its size it is marvellously complete, and well up to date, work appearing in the month of publication having been noticed.

On page 10 the authors state that "caseinogen" is frequently used to designate the form in which casein exists before coagulation; Halliburton, however, applies this term to the proteid whether in solution or precipitated by acids, and reserves the name casein for the curd produced by rennet; lower down a starch-converting enzyme is said to occur in milk, but Béchamp's "galactozymase" from cow's milk is described by him as only liquefying starch paste, and not possessing hydrolytic action; in human milk he describes a true starch-converting enzyme.

The analyses of different kinds of milk on page 11 do not seem quite correct; there is evidently a clerical error in sow's milk; Besana, who has contributed most to the chemistry of ewe's milk, gives a higher percentage of fat than 6·8 per cent.; the proteids of cow's and goat's milk are rather high, and those of human milk rather low as averages.

We are glad to see it recorded that the growth of certain organisms as *B. choleræ* is hampered by the natural organisms of milk.

D'Hout's figure for the fat in skimmed milk (0·02 per cent.) is certainly too low; the authors use "skimmed" as synonymous with "separated."

On page 18, Babcock's method of total solid estimation is erroneously attributed to Richmond.

We are somewhat surprised that on page 19 the quantitative estimation of sulphates should be recommended as a means of detecting watering; few waters contain as much as 0·01 per cent. SO_3 (10 parts per 100,000), and more than that quantity has been found in milk.

The printer does not always manage to place the decimal-point correctly on page 20, where the error due to the extract from Schleicher and Schüll's coils is given as 0·003 per cent. in place of 0·03 per cent.

It is hardly correct to say that Hehner and Richmond's formula was based on perfect processes of fat-extraction, as they used unextracted coils, and made a correction; by a fortunate compensation of errors this formula is near correctness, as shown by many hundreds of analyses. Richmond's slightly-modified formula, which is more scientifically correct, does not seem to be generally known.

A very useful modification of Sebelein's method of casein and albumin estimation is described on page 34.

At the bottom of page 37 it is directed to dissolve mercury in an equal weight of nitric acid in the preparation of Wiley's acid mercuric nitrate; Wiley's directions are to use twice the weight of nitric acid. Vieth's formula for correcting the polarization figures for the volume of the fat is not given.

On page 54 a table is given of the limits adopted in various countries; public analysts will open their eyes at seeing their limit looking miserably small at the bottom of the list.

The chapter on butter is perhaps the most unsatisfactory part of the work, the only method described being the Reichert process; and although this may serve in America, where fine mixtures do not flood the market as in England, it does not alone suffice for the needs of English analysts, who have to grapple with small adulterations.

Probably Drs. Leffmann and Beam have considered methods of distinguishing between proteic, amidic, and ammoniacal nitrogen in cheese as beyond the scope of their book; these are, however, of importance, when the quality of a cheese, apart from its purity, has to be determined.

We would advise every analyst, teacher, and student of milk analysis to obtain this book; it should be widely used as a handbook on the subject, as although it is not free from imperfections, these are mild beside the glaring inaccuracies of the present day text-books. We are inclined to think that the American spelling will somewhat militate against its sale in this country. H. D. R.

Modern Microscopy: A Handbook for Beginners. By M. I. Cross and Martin J. Cole. London: Baillière, Tindall and Cox. Price 2s. 6d.

This work contains much useful information on the choice of a microscope, and will be found of great value to persons who are about to make a selection of such an instrument. The first portion of the book, written by Mr. Cross, is devoted to descriptions (accompanied with excellent illustrations) of several of the instruments and their accessories now before the public, beginning with the simpler forms and proceeding to the more complex. By the aid of these the novice is able to ascertain the form of instrument best suited to his requirements. Plain and simple directions are given for the use of the instrument, and special reference is made to the important subject of illumination, the various condensers being illustrated and their use described. In the second part, which is from the pen of Mr. Cole, who is an old and experienced hand, instructions for cutting, staining, and preparing tissues for examination are succinctly given. These, if carefully followed, cannot fail to lead to the attainment of good results. The work, which is clearly printed and neatly bound, is exceedingly moderate in price. W. J. S.

Experiments upon Magnesia Alba, Quick-lime and other Alcaline Substances. By Joseph Black, M.A. Edinburgh: W. F. Clay.

This little book, which emanates from the Alembic Club of Edinburgh, is a reprint of a paper of Dr. Black's, first published in 1755, copies of which are now scarce and difficult to obtain. As stated in the preface, the objects in reprinting the paper are: "(1) To enable students of the history of chemistry to possess themselves of a copy of this important contribution to the foundation of chemistry as an exact

science ; and (2) to place within the reach of every student of chemistry a model of clear reasoning and of inductive investigation, which is second in this respect to nothing in chemical literature that has appeared in much more recent times." In spelling and punctuation the original has been closely followed. It is to be followed by reprints of the important writings of other British chemists. W. J. S.

CORRESPONDENCE.

To the Editors of THE ANALYST.

DEAR SIRS,—It was a wish to avoid controversial matter that led us to state "Stokes and Bodmer's method is suitable, but probably not very reliable" (ANALYST, xviii. 171) ; we thought that, as we had methods at our disposal that were more satisfactory, it was unnecessary to detail our reasons for thus dismissing it. As Messrs Stokes and Bodmer now take exception to the statement, and accuse us of condemning the Pavy method of titration without trial, merely because Hehner had criticized it adversely, we deem it advisable to give the reasons which we had previously omitted.

We did consider Hehner's results, showing the influence of carbonates and chlorides, as having a direct bearing on the subject ; chlorides to small amount being introduced with the sugar solution, and carbonates being formed by absorption of carbon dioxide from the air by the caustic alkali, both before and after making the solution.

Chittenden (*Studies from the Lab. of Physiolog. Chem., Yale Univ.*, ii. 46) and Ogáda (*Jahr. f. Thierchemie*, xv. 275) have also shown that Pavy was misled by the use of this method.

We, however, recognized that by taking the precaution of standardizing the solution on pure milk and cane sugar (inverted) under the conditions of the experiment, these theoretical objections might be overcome ; an examination of the results given by Messrs. Stokes and Bodmer, in their original paper, where they say that these precautions were taken, convinced us that the method was "probably not very reliable" (for condensed milks).

In that paper the authors give six determinations of known amounts of cane-sugar added to milk ; the amounts added were small, from 1·0 per cent. to 10·5 per cent., and are consequently not comparable directly with the quantity contained in condensed milk. We have, therefore, calculated the error per cent. on the amount taken :

No.	Taken.	Found.	Error Per Cent.
1	1·00	0·95	− 5·0
2	3·25	3·19	− 1·9
3	2·90	2·82	− 2·8
4	3·20	3·05	− 4·7
5	5·50	5·68	+ 3·3
6	10·50	10·80	+ 2·9
		Probable error	± 3·6

Assuming that condensed milk contains an average of 40 per cent. of cane-sugar, the probable error in the determination of this method would be $\pm \frac{3{\cdot}6 \times 40}{100}$, or ± 1·44 per cent.

The limit of accuracy of a method is about 2·7 times the probable error, and therefore the Stokes-Bodmer method of estimating cane-sugar in condensed milk is reliable to 4 per cent.

Being in possession of methods giving greater accuracy than this, we did not feel justified in devoting our time to a critical examination of Pavy's method of titration ; we wish it to be distinctly understood that our criticism was not intended to apply to cases in which the percentage of sugar is small, as it is well known that the method of titration with the substance to be estimated increases in accuracy inversely to the amount of substance in a nearly hyperbolic ratio.

After the detailed reasons we have given we do not see how Messrs. Stokes and Bodmer can object to our very qualified condemnation of their method in reference to condensed milk analysis as "probably not very reliable."—Yours, etc.,

H. DROOP RICHMOND.
L. K. BOSELEY.